NOVEL NON-DESTRUCTIVE MICROWAVE EXTRACTION OF ORGANIC MOLECULES FROM ROCK AND SOIL SAMPLES

Interim Report

JPL Task 1022

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A. OBJECTIVES

Accurate *in-situ* analysis of chemical compounds of astrobiological importance from planetary rocks and soils is strongly dependent on the ability to extract appropriate materials from such samples. In studying the chemical composition of any planetary sample, the most scientifically interesting part of that sample might be inside of it, underneath a protective outer surface. Presently, volatile extraction is limited to the sampling of the surface of a rock or soil from conductive heating or laser ablation. However, in-situ analysis of rock or soil samples for organic molecules would be significantly enhanced by sampling the rock interior, not just its surface.

Cryptoendolithic microorganisms have been demonstrated to survive in the very hostile dry valleys of Antarctic deserts. They accomplish this by finding protected environments inside of rocks where they grow between the rock crystals (Friedman 1982). Protected by a few cm of rock from UV radiation and harsh temperatures of their external environment, they flourish in an environment that may be analogous to ones which may have existed on the Martian surface. This makes rock interiors a prime spot to look for trace elements and microfossils as evidence of extinct Martian life.

The conventional conductive heating method is perhaps the most straightforward of techniques presently available for use by a landed spacecraft. However, it has three major shortcomings: inefficient heating, slow response time and high power loss. Similarly, laser ablation to extract volatiles from soil/rock samples is found to be inefficient since heating of the sample is limited to a small surface area and a very small penetration depth. Therefore, we propose to use microwave energy to extract compounds of interest (such as organic molecules) from the interior of soil and rocks samples. We present this technique (named the Microwave Enhanced Extraction Technique (*MEET*)) as a precision tool to selectively remove compounds of interest by volatilization. This process is useful in delivering controlled heating to the interior of samples, where temperatures can range from just above room temperature (~30ºC) to well over 1000º C. This technique simply offers the best chance to extract specimens from deep inside Martian soil and rock samples. Because microwave energy is deposited directly into the entire sample without the mediation of conductive heating, the microwave heating process is very efficient without an unnecessary power loss, the sample reaches its vaporization point very rapidly (Barnabas et al. 1995), and the extraction process is very energy-efficient (Lopez-Avila et al. 1994). For example, it was reported that extraction of pesticides from soil using less than 1 min of microwave radiation is comparable to more than 1.5 hr of traditional heating, and the quality of the extracts were vastly superior to that obtained by those methods (Granzler et al. 1986).

The objective of this proposal is to demonstrate the feasibility of the MEET technique to sublimate trapped atmospheric gases, chemical species and especially organic molecules in soils that could help elucidate questions on Martian abiotic and biotic chemical evolution. Furthermore, we will explore the utility of changing the frequency of radiation, attempting to raise the temperature of different species one at a time, and vaporizing them so they can be introduced into a mass spectrometer. Some preliminary experiments in a frequency-tuning approach have already been carried out here at JPL, where pure amino acid samples in a microwave cavity have been shown to sublimate at different frequencies. As shown in Fig 1., ~ 10 mg of proline (115 amu) sublimated within a few seconds at a frequency of 2.393 GHz while ~ 10 mg of alanine (89 amu) sublimated at 2.407 GHz (*both with <8 watts of total power*). By frequency tuning other chemicals present, the host matrix will be minimally heated, reducing the amount of heat energy needed.

The proposed proof-of-concept laboratory study will focus on a) the utility of selectively extracting molecules of interest by choosing a discrete microwave wavelength that only sublimates certain compounds while ignoring the rest, and b) exploring and comparing different MEET procedures (with and without solvent added to the sample of interest). This work will dramatically decrease the power and mechanical complexity of sample introduction into a variety of instruments likely to be included on an in-situ astrobiological mission to Mars. Follow up funding from the NASA PIDDP and/or ASTID programs will be sought.

B. PROGRESS AND RESULTS

The experimental setup required use of a fume hood to be able to properly vent the organic material after it had been sublimated, and the laboratory in 183-623 did not have an installed fume hood. Therefore progress on this work has consisted of getting a fume hood installed. This took approximately six months to finish due to a variety of reasons. The hood was approved by the JPL safety office at the end of September 2002, and the experimental apparatus was set up and now is fully operational. Preliminary data from two amino acids, Proline and Alanine, were taken as shown in Fig. 1.

We expect to obtain data from other amino acids as well by the end of October, 2002. This work will be the basis of a planned ASTID proposal for follow-up funding.

C. SIGNIFICANCE OF RESULTS

Work in progress.

D. FINANCIAL STATUS

The total funding for this task was $40,000, of which $20,000 has been expended.

E. PERSONNEL

No other personnel were involved.

F. PUBLICATIONS

N. Budraa, I. Kanik, and L. W. Beegle, "Microwave Heating of Astrobiologically Interesting Samples,"To be submitted, 34th LPSC science conference in Houston TX, March 2003.

G. REFERENCES

None.

H. APPENDIX

Novel Non-Destructive Microwave Extraction of Organic Molecules From Rock and Soil Samples

Fig 1. Pressure vs. microwave cavity frequency for two amino acids, Proline (*left*) and Alanine (*right*), in the same microwave cavity along with structural diagrams.